

Invention was conceived and with due diligence reduced to practice prior to the effective date of the Sharoni reference. (The effective date of publication, i.e., June 4, 2001, can be found at <http://www.iop.org/EJ/toc/0953-8984/13/22>.)

3. This Declaration, and prior invention, is supported by copies of pertinent pages from my laboratory research notebook, entries which were contemporaneously witnessed by Graduate Student Naomi Davis. Date redacted copies of the aforementioned notebook pages are provided collectively as Exhibit A and incorporated herein by reference. These documents establish that the Invention was made at least as early as March 26, 2001, which is a date earlier than the effective date of the Sharoni reference.

I hereby declare that: All statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; that those statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 1001 of Title 18 of the United States Code; and that willful false statements may jeopardize the validity of the Application or any patent issuing thereon.

Date January 13, 2004

David C. Dunand
David C. Dunand

Monday

I discussed last week with D.N. Seidman the idea of superconducting composites based on the new compound HgB_2 . I had mentioned to him the following simple idea:

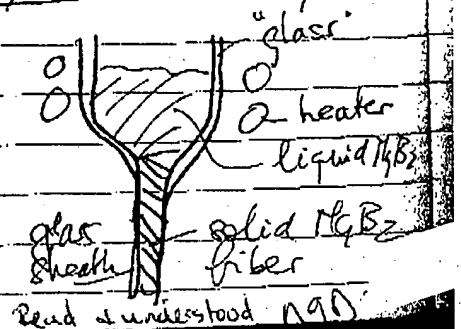
① Infiltrated $\text{Hg}-\text{HgB}_2$ composites

The $\text{Hg}-\text{B}$ phase diagram shows that Hg and HgB_2 are at equilibrium with each other. It should thus be possible to infiltrate a packed bed of HgB_2 powder (which may have been pre-sintered to make a continuous skeleton) with liquid Hg . The net result is a composite with a continuous superconducting phase of HgB_2 embedded in a continuous metallic Hg phase, which allows for thermal management (conducting heat away from HgB_2 and preventing loss of superconductivity) and also being able to carry current if s.c. is lost.

The following other ideas came to me, undisclosed to D.N. Seidman:

② Fabrication of HgB_2 fibers

Superconducting fibers would be very useful embedded in a non s.c. matrix (polymer, metal, glass, ceramic) which provides thermal and mechanical properties. Existing s.c. fibers can be made by drawing (when ductile) or by powder sintering (when brittle). My idea uses a different technique, i.e. the Taylor wire technique. HgB_2 powder is packed in a "glass" tube (any amorphous ceramic such as Pyrex, F glass, etc). The HgB_2 is melted and the glass is quickly drawn into a hollow fiber containing HgB_2 which solidifies into a continuous fiber, which can be spooled.

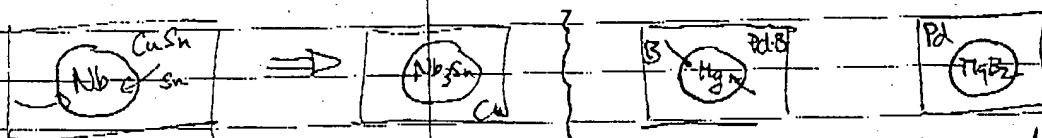


Taylor wires have been made with many metals (see Donald, 1987).

The following requirements are needed: (1) no reaction between glass and MgB_2 , (2) working temperature of glass must be higher than MgB_2 , (3) glass must become highly viscous before MgB_2 solidifies, lest it deforms and breaks the MgB_2 . Pyrex seems a good first choice.

The glass sheathed MgB_2 fiber can then be embedded in a matrix (metal, glass, ceramic, polymer) to form a composite by usual composite processing techniques. Alternatively, the glass can be dissolved by an acid (HF-containing acid) and the bare MgB_2 fibers can be used in a composite. Some dissolution of MgB_2 may be tolerable as part of the glass removal.

- ③ Ductile $Mg-B-Pd$ wires as precursors for $Pd-MgB_2$ composites. Nb_3Sn-Cu composites are ideal, as Cu prevents thermal run-away. They are produced by sticking Nb wires between a Cu-Sn bronze matrix and drawing, resulting in a ductile Cu-Sn/Nb composite which can be drawn and shaped (coiled, etc) to shape. Heat-treatment diffuses Sn to the Nb fibers, forming the brittle Nb_3Sn superconductor in a Cu matrix.

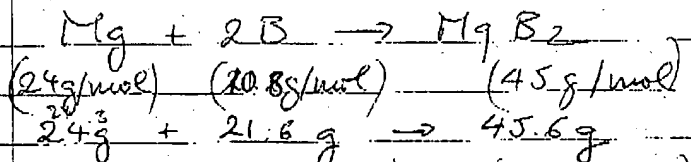


The same idea can be used for MgB_2-X composites, where X is a ductile metal which can be alloyed with B. A good example is Pd, which can accept 2.2 wt% B (at%). So, Mg wires could be embedded within sheets of Pd-B alloy, diffusion-bonded, drawn or shaped as needed. Upon heating below 650°C (melting point of Mg),

Mr. R. Parsons behind pure Pd

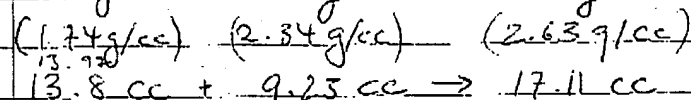
calculations for volume fractions of Pd/MgB₂ composite

MgB₂ synthesis



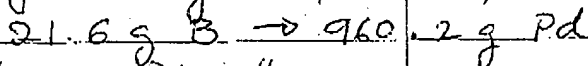
$$\frac{9.23}{13.97+9.23} = 0.398$$

$$\frac{9.23}{17.11} = 0.539$$

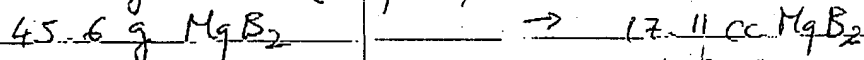
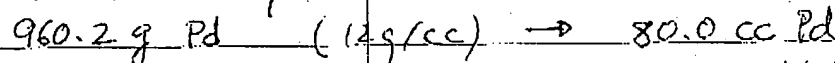


shrinkage: 5.9 cc (27%)

Mg-B alloy Pd-2.2wt% B (max solubility)



Mg-B-Pd alloy



83.4 vol% Pd
17.6 vol% MgB ₂

→ 1/6 of the volume is MgB₂



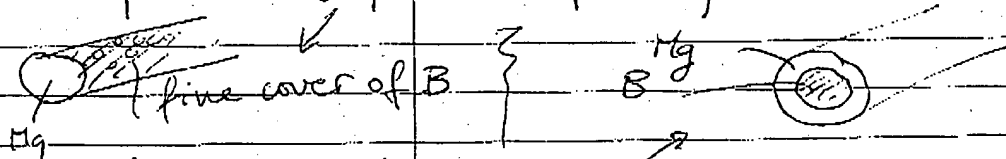
$$\frac{\pi a^2}{4b^2} = 0.176$$

$$\frac{a}{b} = \sqrt{0.176 \frac{4}{\pi}}$$

This volume fraction is not very high, but still commercially feasible, provided price of Pd is not too high.

NB: some Mg will dissolve in Pd

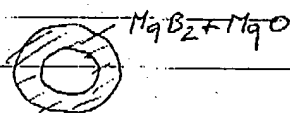
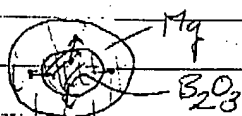
- Additional B could be introduced by dipping Mg wires in B suspension before compacting with Pd or Pd-B foil



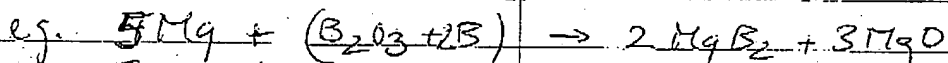
or filling Mg tubes with B powder and then compacting with Pd. Extrusion would still be easy with B powder.
 read + understood Planning. Lamm

- Alternative is to fill Mg tubes with B_2O_3 , which is viscous at extrusion temperatures. The net reaction is then

$$4Mg + B_2O_3 \rightarrow MgB_2 + 3MgO$$



or a suspension of B particles in B_2O_3 , which will remain viscous & drawable.



The advantage is that the metallic matrix can now be a metal different from Pd, and the volume fraction of fibers has no restrictions

MgO_2 fibers (160 μm in ϕ) have been disclosed at the APS meeting. B fiber is exposed to Mg vapors and forms MgB_2 which was found to be superconducting.

Taylor wire technique has following advantages:

- much more rapid production
- diameter easily changed (1-100 μm typically)
- different grain structure
- sheath of glass.

Good matrix for "filled-tube" process: any metal which does not dissolve into Mg (which would contaminate MgB_2)

1. Cu: zero sol. in Mg; eutectic at 48°C; $MgCu_2$ & Mg_2Cu ; 2wt% Mg in Cu
2. Ni: zero sol. in Mg; eutectic at 50°C, $MgNi_2$ & Mg_2Ni ; zero

read & understood,

1/20/91

Solubilities

Metal X	Sol in Hg	Alloy X	Intermetallics	Eutectic (°C)
Cu	0	~3wt%	2	483
Ni	0	0	2	506
Co	0.12	~1.5at%	0	651
Ti	0	0	1	635
Fe	0	0	0	—
Cr	0	0	0	—
Au	0	huge	many	575
Be	0	0	1	—
Pd	0.23	5wt%	7	540
PE	0	0?	5	575
Nb	0	0	0	—
Mo	0	0	0	—

Other advantages:

high conductivity: (Cu) (but reduced by Hg in solid soln)
Au (but β)

Processing: (Cu) low melting
electrodeposition: (Cu, Ni, Cr, Au, Pd, Pt)

no bowdles (Cu), Au
read & understood
Naaman & Lamin

Other Idea:

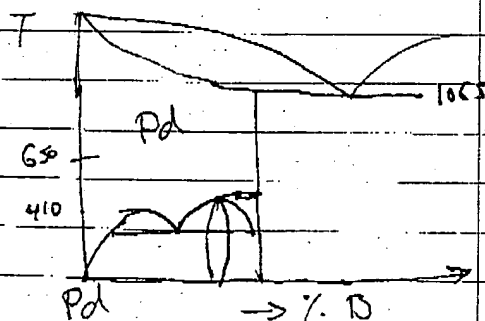
React B with Cu-Mg melt $2B + Cu + Mg \rightarrow MgB_2 + Cu$
 this is only possible if Cu does not dissolve and
 deteriorates MgB_2 s.c. properties

1. infiltrate B fibers or B skeleton
with Cu-Mg melt
2. react in the melt
3. solidifying Cu-MgB₂ composite.

This will work with non-brittle formers, i.e. mostly Cu and Au

Reader understood

Thinning of



pure B dissolves Cu (~16 wt%) at 1013°C

1 mol B = 4.61 cm³ ~ 40 vol%

1/2 mol Mg = 6.89 cm³ ~ 60 vol%

30 vol% B } ~ 50 vol% MgB₂ → 2/3 MgB₂
 45 vol% Mg }
 25 vol% Cu 25 vol% 1/3 Cu

BMG → batch program works! → code
 some cryst. matrix! → new paper with double
 no creep, error due to reloading after
 2 h hold → throw away data
 stand alone f- compression
 paper { - hold
 - matrix info

• MRS paper → draft on Monday
 practice talk on Thursday

Tasker Carr Search Com
 ↳ ↳ carbon steel

Taylor wire MgB_2 trial

- (Went to glass blowing shop and tried with pyrex (10 mm)
- can draw easily empty wire
- MgB_2 powder (~ 1 cm high) gets red hot, but no melting visible
- Can partially draw pyrex, but eventually flakes. MgB_2 does not burn.
- Problem is ϕ is too large, cannot keep the whole block of MgB_2 hot. → try finer tube, maybe try a torch with multiple outlets.

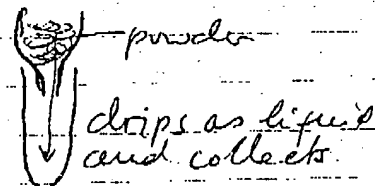
Tried finer wires ~ 4 mm, and after a few trials, got a wire ~ 100-200 μ m, with black MgB_2 inside. It is not clear whether the powder was melted throughout, but it seemed to be

• tried Cu in 4 mm tube (pyrex), but not successful due to high T_m (glass necked before Cu melted)

• Zn powders worked, nice Taylor wires.

• Tried again MgB_2 and got a nice length ~ 10 cm uninterrupted. Pyrex was not cleaned, so bubbles interrupt wire in some parts. Maybe also need to "pre-melt" materials, e.g.

as done for $AgCl$ during thesis (but $T_m = 455^\circ C$ vs $800^\circ C$ for MgB_2)



glass shop closed

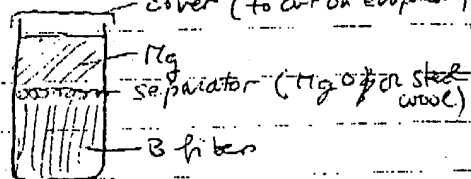
read Campfield paper (PRL 86, 2423) and had the following

idea: integrated fiber formation & composite fabrication

1. Put B fibers and Mg into crucible

2. Heat at $950^\circ C \rightarrow Mg$ melts and seals crucible

hold for 2 hr $\rightarrow Mg$ vapors react with B fibers and form MgB_2 fibers like Campfield's paper



3. Pressurize with gas and force Mg into fiber preform

$\rightarrow MgB_2 - Mg$ Composite

One of the main advantages is that MgB_2 fibers are formed in-situ

They do not have to be handled, bundled, etc, which would

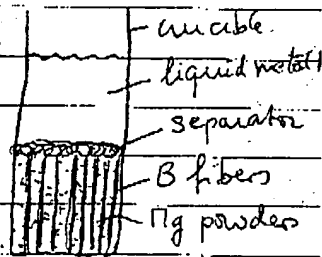
easily damage or break them, because Campfield describes them

Another possibility is to infiltrate B-fibers with molten Mg and keep the composite at 950°C for 1-2 h, long enough for complete reaction to form MgB_2 . Solidifying gives a Mg composite with MgB_2 fibers.

Method to produce MgB_2 metal matrix composites

Canfield's paper shows that MgB_2 fibers can be produced by exposing B fibers to Mg vapors at 950°C for 2 h. However, the fibers are very brittle and bent after fabrication. It will be difficult to bundle them and subsequently infiltrate them without breakage. A solution is to synthesize and infiltrate the fibers in two closely consecutive steps, without handling the fibers.

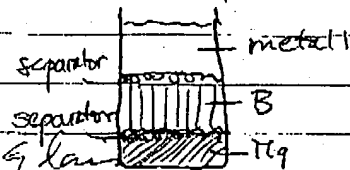
Step 1: Heat-up assembly to synthesis temperature. Mg melts and vaporizes, reacting with B-fibers to form MgB_2 . If the metal 1 is melted, it forms a liquid seal, thus preventing escape of Mg vapors. A non-wetting separator prevents metal 1 from contacting fibers. Examples for metal 1: Mg, Al, Cu alloy with low ~~melting~~ solidus (bronze, brass).



Step 2: pressurize with gas the crucible, forcing liquid metal 1 through the separator and between the MgB_2 fibers, thus making a composite. Solidify and extract composite from crucible. If metal 1 has higher melting point than synthesis temperature of fibers, first raise temperature to melt it.

Complex shapes could be made by this process.

A variation is to put Mg below fibers with separator permeable to Mg vapors.



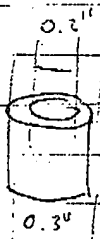
see p. 136: 24 g Mg
+ 21.6 g B

13.8 cc Mg 60 vol %
9.23 cc B ~ 40 vol %

so at best: 20 vol % B

30 vol % Mg powder

50 vol % porosity



$$\phi 5 \text{ mm} \quad A = 19.6 \text{ mm}^2 \rightarrow 3.9 \text{ mm}^2 B$$

0.2"

each fiber is 100 μm $\phi \rightarrow A \pi \cdot 0.05^2 =$

$$0.2 \left(\frac{5}{0.1} \right)^2$$

500 fibers

drop into 0.3" crucible

2 cm \rightarrow 10 m

Observe Dorian run infiltration

1. Turn H₂O on (circulate first at ≈ 125 psi)
open to machine \rightarrow flow switch on
zero pressure transducer
2. Close vessel - slide in
- put PB sheet
- tighten nut until lid is flush with vessel
3. Connect 1 p-transducer (showing as psi x 5, full vac = -65 to 4 TC (check then
4. Open gas tank, set 2nd stage regulator at 150 psi
5. purge 3 times - use valve 1 only
6. check for vac. leak: insulate vessel
verify vac. is constant for 4-5 min
7. Switch on main power, turn to (50%) on both variacs
 \rightarrow upper zone: 5.5 A / 100 V
 \rightarrow lower zone: 4 A / 100 V
wait $\sim 1\frac{1}{2}$ h \rightarrow 350°C
8. put T recorder on turn to (75%) (top only)
 \rightarrow upper zone: 8.5 A / 150 V
wait ~ 45 min \rightarrow turn to (90%) (top only) (70% bottom)
wait ~ 40 min \rightarrow turn down to 50% (both)
and fine-tune
45 min
9. Infiltrate
1. increase to 550 psi (regulator)
2. open valve #2 to vessel
3. switch off power
4. open valve #1 and pressurize to 550 psi
(35 atm)
5. close valves 1 & 2 (will leak $\approx 1-2$ psi/min)
10. Cool down ~ 1 h to 200°C
turn off water, vent (use vent valve)
open

Mg 99.99 / 2 pieces from Emma (26.33g)

started cutting pieces

cut tubing & cleaned tubing

tubes: 0.555" ID = 14.10mm
0.305" ID = 7.75mm

filling in small tube to 2cm: $V = 943 \text{ mm}^3 = 0.943 \text{ cm}^3 \rightarrow$

assuming 50% packing: 0.472 cm^3

$0.472 \text{ cm}^3 \rightarrow 2.63 \text{ g/cc} \rightarrow 1.24 \text{ g MgB}_2$

1.70 $\rightarrow 0.802 \text{ g Mg}$ double

(1.60g Mg)

for large tube, multiply by $\left(\frac{0.555}{0.305}\right)^2 = 3.3$

$\rightarrow 4.1 \text{ g MgB}_2$
 5.3 g Mg

called up

else will send ~ 30 feet of 4 mil B fibers for free

Goodfellow charges \$100 for 10m. [minimum order: 1 lb \$992 for 4 mil \$1159 for 6 mil]

5um W core, 100um diameter, 20.2 mg/m

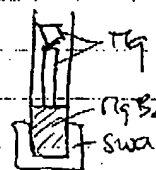
$\rightarrow 10 \text{ m} = 0.202 \text{ g}$

prepare small crucible: Mg: (1.605g) (2 pieces)

add MgB₂ to crucible bottom far: 47.210g

-62g top + push with rod
48.208
383
48.441g

add pieces of Mg on top



$\rightarrow 1.231 \text{ g MgB}_2$

measured height: 6.5mm??
not right, was not "touching bottom" at first!

4.5mm for 0.233g $\rightarrow \sim 22.5 \text{ mm}$ for full mass, OK!

leave for evening

146
Sue Abkowitz

RT
E (ksi) ~~5862~~
Ti64 : 16.88
Ti64+10W: 15.67
↳ (5.609)
-10%

600°F
G (ksi)
~~16.64~~

modules measurement

5.041 → 5.052 → 5.056 → 5.060 → 5.067
0 5 min 10 15 20 min

0.02

change 0.4% in 20 min

- No dissolution: no, T too low, G increases
- W burn off: no, " "
- $\beta \rightarrow \alpha$ transf. is most likely!

(check if Ar is used)

mass: 4.633
volume: $\frac{4.633}{1.5} = 2.355 \text{ cm}^3$
↳ 1.98 g/cm³

Alfa Aesar

prepare large crucible 4.633 g HfB_2 (15 mm height)
5.768 g Zr - 99.99% (2 large pieces, 1 small)

- put a thin ZrO_2 felt layer on top of Hf , stuff crucible to the top w. Al_2O_3 felt (to prevent powder blow out during vac)
- load sample in vessel, contained in S.S. crucible/pot
- evacuate/purge 3 times

11:35 vacuum -76, insulate vessel (leak check)

12:15 up low -75 → 20 K, no leaks

12:18 50% 50% T₁ T₂ T₃ T₄ vac
20.7 -76

12:30 60% 60% 191 -73

1:40 " " 516 -70

2:00 75% 75% 582 568 -70

2:35 65% 65% 742, 743, 743 (734) -69

2:43 735, 737, 739, 735

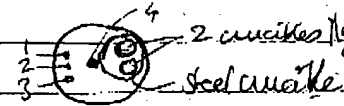
2:49 68% 68% 732

2:55 " " 734, 737, 739, 734 switch off power

2 minute pressurization to 2400 (980 psi)

3:40 depressurize 170°C, 1400 (280 psi)

Mg is at level of T₂ & T₄



new large crucible old Hg B₂ powder from previous expt: 3.475g
new 0.939

old Hg: 4.558g 2 pieces
new Hg: 2.4704 1 piece

7.028g (99.99%)

4.419g
(Alfa Aesar)

0.30 + 2 Hg/
add 0.191g to small crucible

both crucibles leave Hg to the rim, put no felt on top

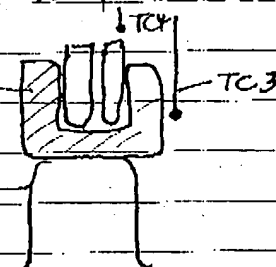
• load crucible in infiltrator

put them in huge graphite crucible
on top of s.s. crucible

much less insulation as last time
should get better heating

TC3 is next to gr. crucible

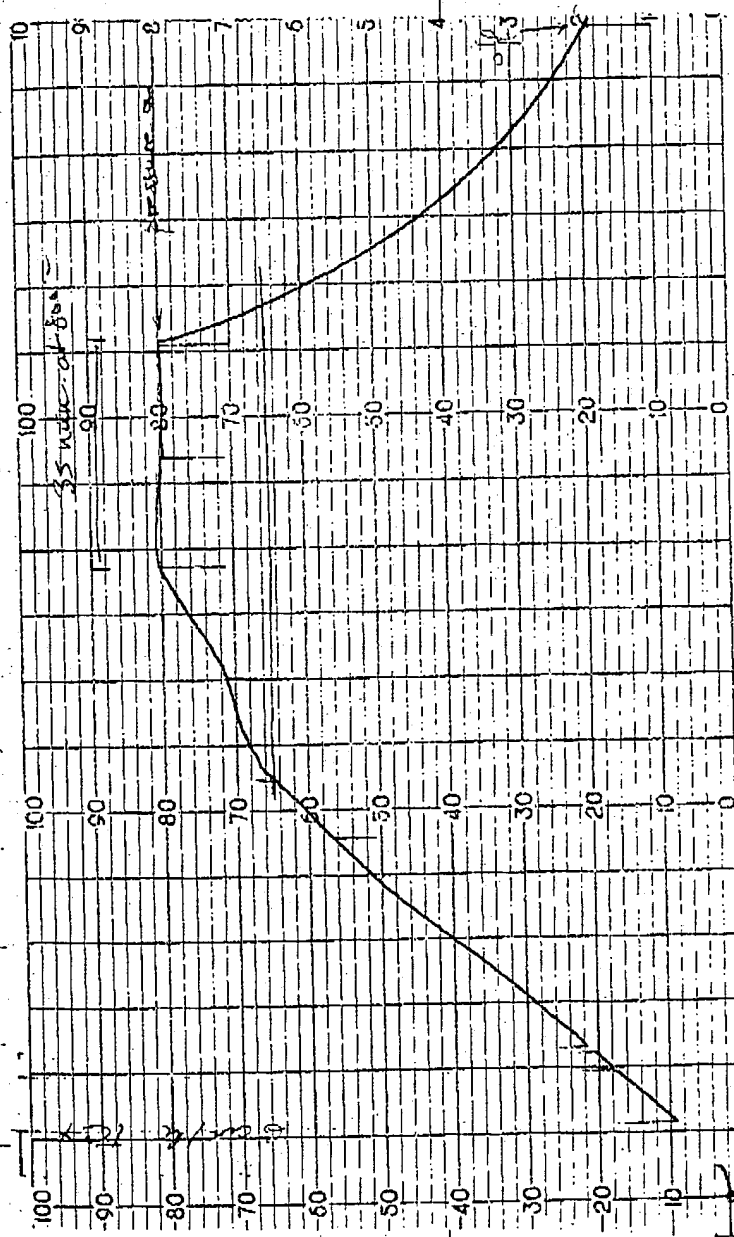
TC4 is just above steel crucibles



• evacuate to -67, insulate & leave for the night 11:35 PM

9³⁰ AM, vac at -41, good cal! Evacuate & flush twice

	P	T ₁	T ₂	T ₃	T ₄	upper lower	lower high	
9:55	-55	24	22	19	21	70%	70%	change Ar bottle
10:25	-55	406	382	330	295	"	80%	
10:47	-53	568	569	590	538	75%	85%	
11:14	-51	688	702	732	712	80	95%	
11:30	-50	797	804	825	801	80%	95%	
11:45	-49	795	795	801	799	"	80%	
12:05	-49				803	"	"	switch off, infiltrate
12:07	2525				755	0	0	in 60 sec
12:10	2232				662			
12:39	1468				286			
12:52	1312				195			pump out, open



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Carbon crucible contains 4.0 g of ~~dark~~ coarse, grey dross
could be Mg evaporated and condensed?

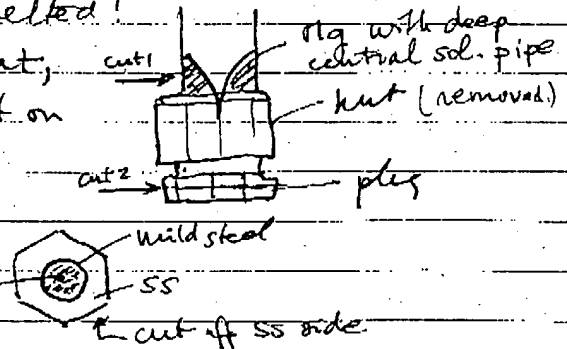
▲ cut large crucible → Mg has melted!

small oxide "hull" left in upper part,
(very light) ^{0.43g} Some white deposit on
top (MgO?). Cut

Cut 1: hack saw, see pipe hole

Cut 2: diamond saw.

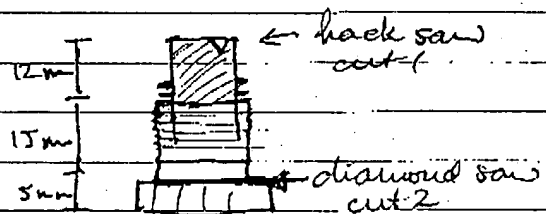
looks infiltrated!



checked under microscope: diamond cut is good enough
to show that the MgB_2 powders are infiltrated
with Mg, forming a Mg/MgB_2 composite.
Took a polaroid, see next page.

▲ open small crucible

• cut 1: Mg to the rim except
small blowhole



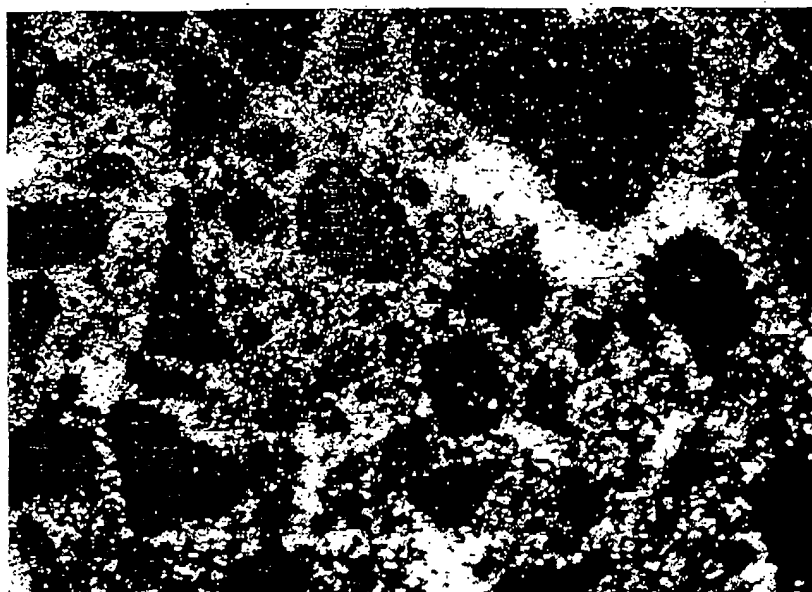
• cut #2 shows only powder, no infiltration

• possible problem: 1) leak at hut

2) bad seal by liquid Mg (unlikely)

3) premature solidification (high surface area)

discard sample.



Mg: white
MgB₂: black

$\sim 1/3$ Mg
 $2/3$ MgB₂

should have
continuous
current
path!

Polaroid: plug side of cut 2, very bottom of sample
sample Mg/MgB₂ (L) 40x objective (400x magnification)

- MgB₂ is agglomerated (size $\sim 50-100 \mu\text{m}$)
- Mg infiltrates as vein between agglomerates (veins: $\sim 10-30 \mu\text{m}$)
- many agglomerates are also filled with Mg ($\sim 1-5 \mu\text{m}$)
- some show holes (pull out due to cutting?)

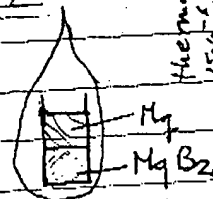
→ this is a fully infiltrated composite, with a few possible regions of porosity (maybe due to closed porosity in original powders or interfering)

To Do: mount & polish this sample
machine steel off
measure conductivity vs. T.

next experiments / possible directions

thermal analysis:
 15% \rightarrow 1,100 \pm 1.5%
 60 \rightarrow 1,500 \pm 2%
 200 \rightarrow 0.1 g/mm

- does Hg wet MgB_2 ?
 use steel crucible to encapsulate



$MgB_2 + Hg$

also may show enhanced sintering due to Hg vapors
 (check density of green compact with the pycnometer)

- react B fibers in capsule, then infiltrate
- " B powders " " "

- infiltrate B fibers and then react in molten state
- " B powders " " "

- infiltrate MgB_2 with Al } new crucibles
- " " " Cu } or BN coated steel

polishing same metallographic section (composite MMC)

1. SiC paper with H₂O

2. 1 μ m diamond in oil (0.05 Al_2O_3 gum up material)

took 2 pictures (see next page)

- composite is successful: 1) full infiltration, no porosity
- 2) no reaction between Hg & MgB_2
- 3) no reaction of either phase w. crucible

Machine shop: asked for EDM of MMC

welding of crucibles

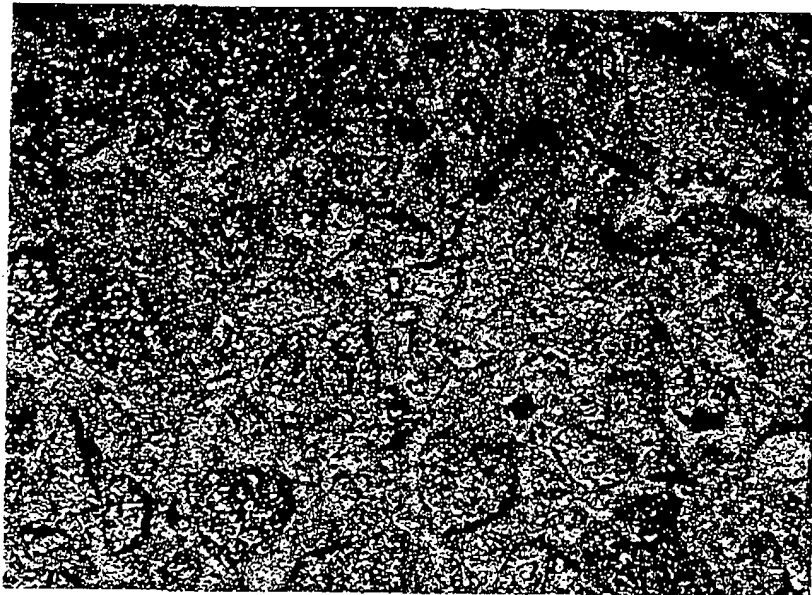
Ordered: B powders, Hg powders, more steel



400x

50 μ m

at the edge of composite, saw
pure Mg anhydride with some
interdendritic structure
not seen in composite



200x

100 μ m

- large (20-100 μ m) Mg B2
- small (2-20 μ m) Mg B2

embodied in Mg
matrix



need to sieve

Metals which can be implanted in current machine

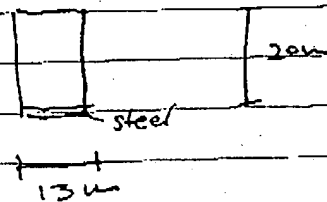
	T_m	T_{ACS}	
Cu	1083 (?)	100%	too hot?
→ Ag	962	108%	
Al	662	65%	
<u>Mg</u>	<u>650</u>	<u>39%</u>	
Zn	420	28%	
Sn	232	16%	
(Brass 260	953 (d))	28%	
" 464	900	26%	
ln	156	20%	
pl	327	8%	
Cd	321	25%	
Au	1064	73%	too hot?

Mutual solubilities in Mg-X systems

	in Mg	in X	intermetallics	eutectic
Mg-Ho	0	0	—	—
Mg-Nb	0	0	—	—
Mg-Co	0	0	MgCo ₂	635
Mg-Cr	0	0	—	no phase diag
Mg-Fe	0	0	—	—
Mg-Be	0	0	1	
Mg-Nd	0	~2% (sat)	4	528
Mg-Pd	0.23%	2.5at%	7	570
Mg-PE	0	0	5	575
Mg-Ti	0.12%	1.5at%	—	651
Mg-Cu	0	6at%	2	483
Mg-Au	0	huge	many	575

mass: 5.213 g

Received machined sample from machine shop. Machinist found composite to be much harder than stainless steel: boride seems well bonded to matrix.



- Side of cylinders show a few pure Hg regions (silvery) but mostly composite
- top part was faced \rightarrow composite (grey)
- bottom part is acut from diamond saw \rightarrow steel composite (black)
- \rightarrow need to cut again with diamond saw

The difference of color (grey for machined composite, black for cut composite) is probably due to smearing of Hg during lathe machining.

- cut bottom slice (1.5 mm) with diamond saw (or 2h!); other side looks nicely infiltrated, except for a large vein of pure Hg. Unlike the other side, it is not black (could be due to corrosion in). Keep slice and mark it with a "B".
- Actually, due to methanol! 1 mm in it produces black color. It is probably etched by it!

- cut upper slice ()

- final dimensions: $h: 17.044 \text{ mm}$
 $D: 12.748 \text{ mm}$
 $m: 4.374 \text{ g}$
 $r = 6.374$
 $V = 2.1753 \text{ cm}^3$
 $\rho = 2.011 \text{ g/cc}$

$$f = 0.63 + (1-f) \cdot 1.74 = 2.011$$

$$f(2.63 - 1.74) = 2.011 - 1.74 = 0.271$$

$$30.5 \text{ vol} \% \text{ HgB}_2$$



other
sides
with
Hg

large regions of pure Hg, possibly displaced by falling chunks of Hg upon loading in crucible. In this region, volume fraction is larger.

**TRANSMITTAL
FORM**

(to be used for all correspondence after initial filing)

Total Number of Pages in This Submission

5

Application Number

10/680,639

Filing Date

October 7, 2003

First Named Inventor

David C. DUNAND

Art Unit

1742

Examiner Name

Ngoclan Thi Mai

Attorney Docket Number

6513-DIV

ENCLOSURES (check all that apply)☐ Fee Transmittal Form☐ Fee Attached☐ Amendment / Reply☐ After Final☐ Affidavits/declaration(s)☐ Extension of Time Request☐ Express Abandonment Request☐ Information Disclosure Statement☐ Certified Copy of Priority Document(s)☐ Reply to Missing Parts/
Incomplete Application☐ Reply to Missing Parts
under 37 CFR1.52 or 1.53☐ Drawing(s)☐ Licensing-related Papers☐ Petition☐ Petition to Convert to a
Provisional Application☐ Power of Attorney, Revocation
Change of Correspondence Address☐ Terminal Disclaimer☐ Request for Refund☐ CD, Number of CD(s) _____☐ Landscape Table on CD☐ After Allowance Communication to TC☐ Appeal Communication to Board
of Appeals and Interferences☐ Appeal Communication to TC
(Appeal Notice, Brief, Reply Brief)☐ Proprietary Information☐ Status Letter☒ Other Enclosure(s)
(please identify below):

Supplemental Response;

Original, executed Declaration of David C.
Dunand;

Return receipt postcard

RemarksAuthorization is hereby given to charge or credit Deposit Account No. 18-0882 for any
fee deficiency or overpayment.

Rodney D. DeKruif

SIGNATURE OF APPLICANT, ATTORNEY, OR AGENT

Firm

Reinhart Boerner Van Deuren s.c.

Signature

Printed Name

Rodney D. DeKruif

Date

January 18, 2005

Reg.
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35,853

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January 18, 2005

This collection of information is required by 37 CFR 1.5. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.11 and 1.14. This collection is estimated to 12 minutes to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.

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Dated: January 18, 2005

BY: Rodney D. DeKruif
Rodney D. DeKruif

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of: David C. Dunand)
Serial No: 10/680,639)
Attorney Docket No. 6513-DIV)
Filed: October 7, 2003)
For: SUPERCONDUCTING)
Mg-MgB₂ AND RELATED)
METAL COMPOSITES)
AND METHODS OF)
PREPARATION)

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

SUPPLEMENTAL RESPONSE

Dear Sir:

As a supplement to the Response filed on January 14, 2005, please enter and consider the original, executed Declaration of David C. Dunand, supporting patentability of the claimed invention. A copy of the same Declaration was previously filed.

Applicant believes no additional fees are required, but authorization is hereby granted to charge Deposit Account No. 18-0882 for any fee deficiency.

This application is now believed to be in condition for allowance. Action consistent therewith is respectfully requested. Thank you for your time and consideration.

Respectfully submitted,

A handwritten signature in cursive script, appearing to read "Rodney D. DeKruif".

Rodney D. DeKruif
Attorney for Applicant
Reg. No. 35,853

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